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**A TECHNIQUE FOR MEASURING
CAPTURE COEFFICIENTS AND
APPLICATION TO THE MEASUREMENT
OF HYDROGEN ADSORPTION BY TITANIUM**

**T. L. Moody
ARO, Inc.**

February 1966

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FOREWORD

The work reported herein was done at the request of the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), under Program Element 65402234.

The results of the research presented were obtained by ARO, Inc. (a subsidiary of Sverdrup and Parcel, Inc.), contract operator of the AEDC, AFSC, Arnold Air Force Station, Tennessee, under Contract AF 40(600)-1200. The research was conducted from May to August, 1964, under ARO Project No. SW3417, and the manuscript was submitted for publication on October 15, 1965.

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This technical report has been reviewed and is approved.

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ABSTRACT

The problems encountered in making capture coefficient measurements are reviewed, and a new experimental technique, employing a rotating gage, is presented. Utilizing this technique, the fraction of impinging hydrogen molecules removed by a continuously deposited titanium surface was determined as a function of the titanium evaporation rate and gas flux. The results demonstrate that there is little to be gained by evaporating more than one atom of titanium for each atom of hydrogen.

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NOMENCLATURE

| | |
|--------------------------------|---|
| A | Area of pumping surface, cm ² |
| a | Area of gage opening, cm ² |
| C | Capture coefficient |
| $\frac{dv}{dt}$ | Volumetric rate of impinging molecules |
| $\frac{dn}{dt}$ | Gas flux, molecules/sec |
| $\left(\frac{dn}{dt}\right)_i$ | Impinging gas flux, molecules/sec |
| $\left(\frac{dn}{dt}\right)_r$ | Rebounding gas flux, molecules/sec |
| G | Geometry factor |
| I | Ion current |
| I _e | Electron emission current |
| I _i | Ion current produced by impinging gas molecules |
| I _r | Ion current produced by rebounding gas molecules |
| K | N _g /N _T |
| \hat{k} | Constant of proportionality |
| M | Molecular weight, gm/gm-mole |
| N | Maximum number of sorption sites per unit area |
| N _g | Number of sorption sites used by a gas atom |
| N _O | Avogadro's number |
| N _T | Number of sorption sites furnished by one titanium atom |
| n _t | Total number of molecules that enter through solid angle ω |
| n _z | Number of molecules impinging from the direction of the normal |

| | |
|-----------------|---|
| P | Pressure, torr |
| P _C | Cell pressure, torr |
| P _g | Gage pressure, torr |
| P _{gi} | Pressure in gage caused by impinging molecules, torr |
| P _{gr} | Pressure in gage caused by rebounding molecules, torr |
| P _i | Impinging pressure, torr |
| P _r | Rebounding pressure, torr |
| Q | Gas flow rate, torr-liters/sec |
| R | Universal gas constant |
| S | Pumping speed, liters/sec |
| T | Gas temperature, °K |
| T _C | Cell temperature, °K |
| T _g | Gage temperature, °K |
| T _i | Temperature of impinging gas |
| T _r | Temperature of rebounding gas |
| α | Ratio of titanium deposition rate to gas impinging rate |
| β | $\frac{\alpha N_{TC}}{N}$ |
| γ | The percent of total number of molecules which enter the gage through a cone of solid angle ω |
| η | Number of molecules leaving point A |
| η _B | Number of molecules arriving at point B |
| η _Z | Number of molecules which leave normal to surface |
| θ | Angle to normal at which molecules strike |
| ξ | $\left(\frac{N}{C} - N_g\right)\left(1 - \frac{\alpha N_T}{N}\right)\frac{C}{N}$ |
| ρ | Radius of sphere |
| σ | Distance between two points on sphere |
| φ | Angle at which molecules leave surface |
| ψ | Probability of nth molecule being removed (n = 1, 2, 3...) |
| ω | Solid angle of cone |

SECTION I INTRODUCTION

In the field of space simulation, large pumping speeds are often required to maintain an environmental test chamber at low pressure. Cryopumps are widely employed for this purpose; however, they are selective in the species of gases that they will efficiently pump. Gases such as hydrogen require such low cryosurface temperatures that cryopumping is not usually economically feasible. As a result, the use of materials that adsorb those gases which are difficult to condense has received considerable interest. For example, titanium has been found to be a useful sorbent for hydrogen (Refs. 1 to 4). Most of the work in this area has been qualitative in nature and only demonstrated the pumping phenomena. However, as part of a general investigation of vacuum production at the Arnold Engineering Development Center's Aerospace Environmental Facility (AEF), Kindall (Ref. 3) has determined the influence of several factors on the capture of hydrogen atoms by a titanium surface.

For practical application to large space chambers, an adsorption pump would require a continuous supply of the sorbent material to maintain a constant pumping speed, since the active molecules in the sorbent surface are being covered with gas molecules. With titanium, this can be achieved by heating a supply of the metal to a temperature near its melting point and continuously evaporating it onto a suitable substrate. Such an approach has already been employed in an existing space simulation chamber (Ref. 4); but, since the pumping phenomena is still not well understood, the work begun by Kindall at AEDC has been extended to attempt to study and refine the technique of continuously pumping hydrogen by titanium.

Originally, the present study was initiated to determine experimentally how the effective capture coefficient of a titanium surface varied with the evaporation rate of titanium and hydrogen gas flux. However, the difficulties encountered in making the measurement led to the development of an improved experimental technique for measuring capture coefficient. The object of this report is, thus, two-fold: (1) The first part of the report is devoted to a discussion of a new experimental technique for measuring capture coefficients, and (2) the last part of the report describes an application of this technique to titanium adsorption.

SECTION II

EXPERIMENTAL MEASUREMENT TECHNIQUES

In all of the analyses or mathematical derivations in this report, it is assumed that the gas is in equilibrium and possesses all of the properties commonly assumed in the kinetic theory of gases.

2.1 CALIBRATED FLOW METHOD

A conventional method of making capture coefficient measurements in the free molecular region is by use of the calibrated flow technique (Ref. 5). With this method, the gas to be pumped is bled into the system at a known rate, Q ; then, when the system has reached steady state, the cell pressure is measured by a suitable pressure gage, and the volumetric pumping speed determined by the expression (Ref. 6):

$$S = \frac{Q}{P_c} \quad (1)$$

The volumetric rate, dv/dt , of impinging gas molecules which strike a surface in free molecular flow is given by kinetic theory as

$$\frac{dv}{dt} = A \sqrt{\frac{RT}{2\pi M}} \quad (2)$$

The capture coefficient, C , will then be given by

$$C = \frac{S}{dv/dt} \quad (3)$$

This method has several inherent disadvantages:

1. The flow rate of the test gas must be calibrated.
2. The deposition rate of the sorbent must be uniform over the substrate area.
3. Gas impurities in the test gas and those caused by outgassing must be accounted for in the pressure measurement reading.
4. The substrate area must be known.
5. The pressure gage must be calibrated for a given gas and gas temperature.
6. The variations in the sensitivity of the pressure gage must be known.

As a result of these experimental difficulties, an improved method of determining capture coefficients was investigated.

2.2 ROTATING GAGE METHOD

2.2.1 Infinite Pumping Plane

Consider now an infinite pumping plane, Fig. 1, with an associated capture coefficient, C . Situated in front of this plane is a rotatable pressure gage enclosed in its own envelope. The envelope has a tubular port of area a on one side only so that it measures pressure only from one direction. At equilibrium conditions it is assumed that a uniform diffuse gas flux, dn/dt , impinges on the pumping surface. From kinetic theory it may be shown that such an ideal gas with a Maxwellian velocity distribution will produce an impinging pressure in the gage of

$$P_i = \frac{dn}{dt} \frac{\sqrt{2 \pi M R T}}{N_o a} \quad (4)$$

when the gage opening is directed away from the pumping surface.

At the pumping surface, gas will be removed at the rate $C \, dn/dt$ and rebounded at the rate $(1 - C) \, dn/dt$. Further, it is assumed that the gage envelope is sufficiently small that its shadowing effect for impinging molecules may be neglected. In this case, when the gage is positioned in the direction facing the pumping surface, it will read a pressure caused by the rebounding gas flux of

$$P_r = (1 - C) \frac{\frac{dn}{dt} \sqrt{2 \pi M R T}}{N_o a} \quad (5)$$

The ratio of the two pressure gage readings will then provide a measure of the capture coefficient:

$$\frac{P_r}{P_i} = 1 - C \quad (6)$$

Equation (6) was derived by assuming that the surface and gas temperatures were the same. However, in Appendix I it is shown that this is an unnecessary requirement.

The application of a rotatable pressure gage to measure the capture coefficient for an idealized infinite pumping plane has served to demonstrate the method; in practice, such an ideal geometry can only be approximated. More practical geometries can be used, but if the geometry chosen for the pumping surface is concave, then multiple collision with the surface must be taken into account. The next two sections describe how this measuring technique may be applied to other types of pumping surfaces.

2.2.2 Hemispherical Pumping Surface

The rotating gage technique is particularly applicable for measuring capture coefficients when a hemispherical pumping surface is used. This can be demonstrated by considering a spherical vacuum chamber in which one half is made a uniform pumping surface; the rotatable gage is located in the plane dividing the pumping surface from the nonpumping surface.

If it is assumed that the gas source into the cell is distributed uniformly over the nonpumping half of the cell, and all surfaces in the cell are assumed to be Lambert surfaces, then the gas flux to and from the pumping surface will be uniform. This is shown by the aid of Fig. 2. If η_z gas molecules per unit area leave the cell surface at some point A, then the number of gas molecules per unit area η_B that arrive at any other point, B, in the cell will be

$$\eta_B = \frac{\eta_z \cos^2 \phi}{\sigma^2} \quad (7)$$

where

η_z = number of molecules that leaves at a direction normal to A

σ = distance from A to B

ϕ = angle between the normal and σ

But

$$\cos \phi = \frac{1/2 \sigma}{\rho} \quad (8)$$

Then

$$\eta_B = \frac{\eta_z \cos^2 \phi}{\sigma^2} = \frac{\eta_z}{4\rho^2} \quad (9)$$

where

ρ = radius of the sphere.

Hence, the impinging flux is uniform over the sphere.

The impinging gas flux, $(dn/dt)_i$, and rebounding flux, $(dn/dt)_r$, are uniformly distributed across the midplane by the following reasoning. If the chamber is at equilibrium with no gas sources or sinks, the spatial distribution throughout the chamber is uniform. If one half of the chamber is made a uniform source and the other a uniform sink, then the spatial distribution from either across the midplane is unchanged. The impinging gas flux across the midplane will give rise to a pressure in the gage of

$$P_i = \left(\frac{dn}{dt} \right)_i \frac{\sqrt{2 \pi M R T}}{N_o a} \quad (10)$$

At the pumping surface, a fraction C of the impinging molecules will be removed and $(1 - C)$ rebounded, but of these only one-half will cross the midplane since, as shown, a molecule leaving the surface has the same probability of striking any other point in the cell. The flux which transverses the midplane will be

$$0.5 (1 - C) \left(\frac{dn}{dt} \right)_i \quad (11)$$

and the fraction which strikes the pumping surface for a second time will be

$$0.5 (1 - C) \quad (12)$$

Of these, $(1 - C)$ will be rebounded so that the fraction of the flux which rebounds after a second collision will be

$$0.5 (1 - C)^2 \quad (13)$$

But of these, only one-half can transverse the midplane

$$\left(\frac{dn}{dt} \right)_i (1 - C)^2 (0.5)^2 \quad (14)$$

and the total flux which transverses the midplane will be

$$\left(\frac{dn}{dt} \right)_r = \left(\frac{dn}{dt} \right)_i \left[(1 - C) 0.5 + (1 - C)^2 (0.5)^2 + \dots + (1 - C)^n (0.5)^n \right] \quad (15)$$

$$= \frac{\left(\frac{dn}{dt} \right)_i (1 - C) 0.5}{1 - (1 - C) 0.5} \quad (16)$$

$$= \frac{(1 - C)}{(1 + C)} \left(\frac{dn}{dt} \right)_i \quad (17)$$

This will produce a pressure in the gage of

$$P_r = \left(\frac{dn}{dt} \right)_i \frac{(1 - C)}{(1 + C)} \frac{\sqrt{2 \pi MRT}}{N_o a} \quad (18)$$

The ratio of the two gage readings will be

$$\frac{P_r}{P_i} = \frac{1 - C}{1 + C} \quad (19)$$

so that the pressure ratio is only a function of the capture coefficient.

It may be difficult to achieve uniform particle flux across the midplane of a sphere, which is a requirement for the validity of Eq. (19). A more practical experimental arrangement for measuring the capture coefficient of various pumping surfaces using the proposed rotating gage method would be to place a hemispherical pumping surface in a much

larger vacuum chamber.* The test gas should be introduced in such a manner that it is reflected by the chamber wall sufficiently to become diffused before transversing the entrance plane of the hemisphere. If the rotating gage is located in the entrance plane of the hemisphere, then the pressure ratio will be related to the capture coefficient by Eq. (19).

It should be noted that the gage will intercept the impinging gas flux and would perhaps introduce nonuniformities. This effect can be minimized by placing the body of the gage outside the hemisphere as shown in Fig. 3.

It has been shown in Appendix I that the gas temperature will not affect the capture coefficient measurement, that is, the ratio given by the pressure gages is insensitive to gas temperature. However, if the gas temperature changes as a result of a collision, this may change the capture coefficient for subsequent collisions. This can be seen by re-writing Eq. (16) in the following form:

$$\begin{aligned} \left(\frac{dn}{dt}\right)_r = & \left(\frac{dn}{dt}\right)_i (1 - C) (0.5) + \left(\frac{dn}{dt}\right)_i (1 - C_2)^2 (0.5)^2 \\ & + \left(\frac{dn}{dt}\right)_i (1 - C_3)^3 (0.5)^3 + \cdots + \left(\frac{dn}{dt}\right)_i (1 - C_n)^n (0.5)^n \end{aligned} \quad (20)$$

The first term is the flux of molecules sampled by the gage after one collision. The second term is the number sampled by the gage which have had two collisions, but the temperature of these molecules may change because of the first collision with the substrate. Since the capture coefficient is possibly a function of gas temperature, it may have a different value. This is indicated by a subscript, 2. Likewise, the remaining terms have subscripts to indicate the possible difference in values for C. This complication did not arise with the infinite pumping plane but is a consequence of having a concave pumping surface and multiple collisions.

It may be possible to evaluate the effect of temperature on the capture coefficient for some of the limiting cases listed below:

1. If the accommodation coefficient is low or if the capture coefficient is not a strong function of gas temperature, then the error in Eq. (19) will be small.

*The convenience of a hemispherical pumping surface only applies to the measurement of capture coefficient.

2. If the capture coefficient is large, then multiple collisions will be negligible; that is, only the first term will contribute significantly to the series given by Eq. (20).
3. If the accommodation coefficient is sufficiently high that it can be assumed that after one collision the gas accommodates to the substrate temperature, then Eq. (19) can be corrected as follows: Make a first run with the substrate and gas at the same temperature and determine the value of C from Eq. (15). Then, make a second run at the desired gas temperature and using the above value of C for quantities such as C_2 , C_3 , and C_4 , determine C_1 .

If the accommodation coefficient and capture coefficient are in the intermediate range, then it will be necessary to know the accommodation coefficient to obtain accurate results.

2.2.3 Other Pumping Surface Geometries

Other pumping surface geometries may be more practical from an experimental standpoint, but they may not lend themselves to the simple analytical treatment as do the infinite plane and hemispherical surfaces. The relationship between the rotatable pressure gage readings and the capture coefficient of these more complex surfaces can be obtained from a numerical analysis by use of the Monte Carlo method (Ref. 7). The essentials of this type of solution are outlined below.

As gas molecules enter the cell through the gas inlet, they are assumed to leave the inlet with a spatial cosine distribution. Also, the mean free paths of the molecules are assumed to be larger than the dimensions of the cell. The computer then calculates the trajectory of each molecule until it intercepts the cell surface. Then, assuming that all surfaces in the cell are Lambert surfaces (diffuse reflection), the computer, by use of a random number generator, calculates the trajectory along which the molecule rebounds and the location of its next collision with the cell surface. This process continues until the molecule strikes a pumping surface. The computer then, from a preassigned capture coefficient, determines the probability of the molecule being removed. The computer accomplishes this by generating a random number from 0 to 1. If the number is greater than the assigned capture coefficient, the molecule is rebounded and the process continues. If the number is less than the capture coefficient, the molecule is removed, and a new molecule is started at the gas inlet. Each molecule that traverses the preassigned location of the gage in passing to the pumping surface is counted. The same is done for molecules passing from the pumping surface. The ratio of these two numbers is then the same ratio that would be obtained by the rotating gage.

It should be pointed out that the Monte Carlo method is purely statistical, and the accuracy of the results depends on tracing enough molecules to obtain a good statistical average.

This technique has been used to handle the case of a cylindrical chamber which was employed in the experimental portion of the work described here. Results of these calculations are given in Section 4.3.

2.3 COMPARISON OF METHODS

The rotating gage method of measuring capture coefficient has several advantages:

1. It does not require a calibrated flow system and allows the measurement to be made at any desired pressure in the free molecular flow regime.
2. Other pumping surfaces, such as shields for evaporation sources, which might become coated with titanium will have little or no effect if properly located (i. e., so as not to interfere with the gas flux entering the gage).
3. During the measurement, it is not necessary to isolate additional pumps from the chamber. As a result these pumps can be used to prevent accumulation of nonabsorbable gases during the measurement.
4. The substrate area need not be known as long as the gage views only the substrate. Also, the deposition only needs to be uniform over the areas from which the rebounding gas flux can enter the gage.
5. It is not necessary that the ionization gage be calibrated, nor will a change in gage sensitivity affect the measurement if a linear ionization gage is used. This is shown in Appendix II.
6. The capture coefficient measurement is independent of gas temperature. (See Appendix I.)

Although the rotating gage technique has several advantages over the calibrated flow method, there are experimental difficulties associated with both methods.

It has been assumed that the flux to and from the pumping surface was uniformly diffuse. In practice this ideal condition may be difficult to achieve, since the flux will be influenced by the cell's contents (gage and shields, for example), location of gas source, and pump inlets. All

of these things will contribute to nonuniformities in the flux, i. e., pressure gradients.

Secondly, when a concave pumping surface is used, multiple collisions with the pumping surface will occur. As pointed out in the discussion for the hemispherical pumping surface, if the gas and surface temperature are different, the gas temperature may be changed after a collision and, as a result, could change the capture coefficient.

When evaporated metal films are used as a pumping surface, the requirement for a uniform pumping surface becomes extremely difficult to realize. In addition, to confine the evaporation to the pumping surface will usually require the use of evaporation shields, which will also become pumping surfaces. However, with the rotating gage technique this problem is reduced as pointed out in the fourth advantage above.

SECTION III THEORY FOR CONTINUOUS TITANIUM ADSORPTION PUMPING

As the titanium condenses on the substrate, a certain number of sorption sites will become available. The probability of an impinging gas molecule finding the sorption site, coupled with the probability of sorption at the site, will be termed the effective capture coefficient; the probability of sorption at the site will simply be termed the capture coefficient. It can be seen that the effective capture coefficient will not only be a function of evaporation rate, but also of the gas flux to the surface.

In deriving an expression for effective capture coefficient as a function of the evaporation rate and gas flux, it will be assumed that the sorption process is purely a surface phenomenon and that each titanium atom will furnish a certain number of sorption sites, N_T . Let N_g be the number of sites used by a gas atom, and let α be the ratio of titanium deposition rate to gas impinging rate.

The probability $\psi_1(\alpha, C)$ of an impinging molecule being removed by a surface completely covered with titanium will simply be the capture coefficient, C .

$$\psi_1(\alpha, C) = C \quad (21)$$

The second molecule which arrives at the surface will have a probability $\psi_2(\alpha, C)$ of being removed. This is the ratio of the remaining

active sites to the maximum number of sites, N , available in a unit area, multiplied by the capture coefficient. The number of active sites available, then, for the second molecule will be the number of sites available for the previous impinging gas molecule, $(\psi_1 N)/C$ minus the number of sites, $\psi_1 N_g$, used by the previous molecule plus the number of sites furnished by the titanium atoms deposited times the number of sites furnished per atom, αN_T , times the probability of hitting a used site,

$$\left[N - \left(\frac{\psi_1 N}{C} - \psi_1 N_g \right) \right] / N$$

The probability of the second molecule being removed is, then,

$$\psi_2(\alpha, C) = \left\{ \psi_1 \frac{N}{C} - N_g \psi_1 + \alpha N_T \frac{\left[N - \left(\frac{\psi_1 N}{C} - N_g \psi_1 \right) \right]}{N} \right\} \frac{C}{N} \quad (22)$$

In the same manner, the probability $\psi_3(\alpha, C)$ for the third molecule which strikes the wall being removed will be

$$\psi_3(\alpha, C) = \left\{ \psi_2 \frac{N}{C} - N_g \psi_2 + \alpha N_T \frac{\left[N - \left(\frac{\psi_2 N}{C} - N_g \psi_2 \right) \right]}{N} \right\} \frac{C}{N} \quad (23)$$

The probability $\psi_n(\alpha, C)$ of the n th impinging molecule being removed will be

$$\psi_n(\alpha, C) = \left\{ \psi_{n-1} \frac{N}{C} - N_g \psi_{n-1} + \alpha N_T \frac{\left[N - \left(\frac{\psi_{n-1} N}{C} - N_g \psi_{n-1} \right) \right]}{N} \right\} \frac{C}{N} \quad (24)$$

Rearranging,

$$\psi_n(\alpha, C) = \left[\psi_{n-1} \left(\frac{N}{C} - N_g \right) \left(1 - \frac{\alpha N_T}{N} + \alpha N_T \right) \right] \frac{C}{N} \quad (25)$$

For convenience let

$$\xi = \left[\left(\frac{N}{C} - N_g \right) \left(1 - \frac{\alpha N_T}{N} \right) \right] \frac{C}{N} \quad (26)$$

and

$$\beta = \frac{\alpha N_T C}{N} \quad (27)$$

Then,

$$\psi_n = \xi \psi_{n-1} + \beta \quad (28)$$

Thus, the first few terms are:

$$\psi_1 = C \quad (29)$$

$$\psi_2 = \xi C + \beta \quad (30)$$

$$\psi_3 = \xi^2 C + \xi \beta + \beta \quad (31)$$

and in general, Eq. (28) may be written,

$$\psi_n = \xi^{n-1} C + \beta \sum_{N'=0}^{N'=n-2} \xi^{N'} \quad (32)$$

where N' is the summation index. The limit of this sequence will then be the steady-state probability $\psi(\alpha, C)$ of a molecule being removed, i. e., the effective capture coefficient. The limit of the sequence yields an infinite series

$$\psi(\alpha, C) = \lim_{n \rightarrow \infty} \left(\xi^{n-1} C + \beta \sum_{N'=0}^{N'=n-2} \xi^{N'} \right) \quad (33)$$

Since

$$0 < C < 1$$

N is very, very large

N_T and N_g are near unity

and

$$\alpha < N$$

then ξ and β are certainly less than one but greater than zero. The first term will converge to zero in the limit and the second will converge to

$$\psi(\alpha, C) = \frac{\beta}{1 - \xi} \quad (34)$$

and N_g/N_T will be the number of gas molecules adsorbed per titanium atom. Let

$$K = N_g/N_T \quad (35)$$

and since N is a very large number, then

$$\frac{\alpha C N_g}{N} < C N_g/N_T + \alpha \quad (36)$$

so that

$$\psi(\alpha, C) = \frac{\alpha C}{KC + \alpha} \quad (37)$$

Equation (37) gives the probability for the capture of a gas particle by a titanium film which is being continuously deposited in terms of (1) the capture coefficient of a titanium film C , (2) the ratio of the titanium evaporation rate to gas impingement rate, α , and (3) the number of gas molecules adsorbed by each titanium atom, K . A method for determining these parameters is discussed in Section VI.

SECTION IV EXPERIMENTAL INVESTIGATION

4.1 APPARATUS

4.1.1 Vacuum Cell and Pumping System

The vacuum chamber used in this study (Fig. 4) was 24 in. in diameter and 18 in. high. Its pumping system was composed of a 6-in. oil diffusion pump backed by a 2-in. oil diffusion pump and a mechanical forepump. The pumping system was separated from the chamber by a 6-in. sliding gate valve. Hydrogen gas at near 300°K was admitted to the cell through a variable leak.

4.1.2 Titanium Evaporation Source

The titanium evaporation source was constructed by twisting ten 20-mil tungsten wires 10 in. in length together. These were then wrapped with 100 in. of 20-mil titanium wire. The source was positioned along the vertical axis of the cylindrical chamber, equidistant from the top and bottom. Evaporation was achieved by resistance heating using the filament as a resistance element and passing a 60-cps current of about 180 amp through it. It was desired that the evaporated titanium be deposited only on one-half of the cylinder. This was accomplished by use of a shield which was a half-cylinder 10 in. long and 1-1/2 in. in diameter positioned so that the titanium filament was located along its vertical axis. The source shield and gage locations are shown schematically in Fig. 5.

It was found that until the substrate was completely covered with titanium the measured capture coefficient would vary slightly for the same evaporation rate and gas load. The effect was small, but it was desirable to eliminate this variation so that consistent results could be obtained. This was done by precoating the surface with another titanium evaporation source. This evaporation source was shielded and constructed the same as the primary source with the exception that it was only 6 in. in length. It was located parallel to the vertical axis of the cell and 1 in. away.

4.1.3 Measurement of Evaporation Rate

The evaporation rate was measured with a thickness monitor and controlled by a rate control unit. Briefly, these instruments operate as follows: A small transistorized crystal oscillator is exposed to the

evaporation source so that the evaporated material is condensed on the crystal of the oscillator. When this occurs, the resonant frequency of the oscillator will change in such a way as to be directly proportional to the mass deposited. This change in resonant frequency is then displayed on the thickness monitor. The same information is also fed to the rate control unit, which displays the rate of the deposition. With this instrument, it is also possible to preset a desired evaporation rate. When this is done, the rate control unit, after sampling the input rate, feeds an appropriate signal to the input of a current transformer, which supplies the necessary power to the evaporation source. A schematic of this system is given in Fig. 6.

The arrangement of the titanium source precluded a uniform evaporation rate over the entire half of the test chamber. However, it is shown in Appendix III that for the ionization gage used in this experiment, 90 percent of the molecules which enter the gage come from within a cone of 65 deg. It is believed that by judicious positioning of the gage and source, the deposition could be made uniform over the area from which most of the rebounding gas flux (which entered the gage) came.

4.1.4 Stainless Steel Cold Surface

During the titanium deposition period large amounts of methane gas appeared in the cell. It was believed that this gas came from both outgassing of the vacuum cell, caused by the hot titanium filament (Ref. 8), and reaction of hydrogen gas with carbon impurities in the filament (Ref. 9).

Methane gas is not appreciably adsorbed by titanium and will not, therefore, occupy a large number of the titanium absorption sites. However, if the methane pressure is of the same order of magnitude or greater than the hydrogen test gas, this would render the pressure measurements of the ionization gage unreliable. However, Davis (Ref. 10) has found that methane gas is readily removed by a stainless steel surface when cooled to liquid nitrogen temperatures.

To remove the methane gas load in this experiment, a stainless steel plate 20 in. in diameter was installed in the bottom of the vacuum cell. In the center of the plate, a hole 6 in. in diameter was cut to allow gas from the cell to enter the diffusion pump. A 3/8-in. copper tube was soldered on the bottom of the plate to carry the liquid nitrogen. This detail has been omitted from Fig. 4 for purposes of clarity. The remainder of the chamber walls, including most of the titanium pumping surface, were at room temperature (near 300°K).

4.1.5 Ionization Gage

The pressure was measured with a Bayard Alpert-type ionization gage. It was mounted on a rotatable feedthrough in such a position that it could be rotated 180 deg about the center of the cylinder (see Fig. 5). When in one position it would view only the substrate area; then, after 180-deg rotation, it would view only the nonpumping surface area. The tubulation of the gage had a length-to-diameter ratio of 2.7.

4.2 TEST PROCEDURE

Each experimental run began by pumping the cell to a pressure in the 10^{-8} torr region by means of the diffusion pumps. Except for the cooled methane trap, the chamber walls remained near 300°K. After the methane trap had cooled to liquid-nitrogen temperature, the substrate was precoated with titanium by evaporation from the 6-in. filament. Next, the rate control unit was set to give the desired evaporation from the 10-in. filament. After evaporation from the 10-in. filament had started, 300°K hydrogen gas was admitted to the cell through the variable leak. The leak rate was adjusted to give the desired gas flux to the pumping surface. This resulted in a cell pressure in the 10^{-5} or 10^{-6} torr region.

The pressure gage was rotated to view only the half of the cell that was not coated with titanium, and the pressure recorded. The gage was then rotated 180 deg to view only the half of the cell coated with titanium, and the pressure again recorded. In addition to these two pressure readings, the evaporation rate was also recorded. The rate control unit was next adjusted to give a different evaporation rate, and the same procedure repeated.

4.3 DATA ANALYSIS

Because the geometry of the cylindrical test chamber does not lend itself to analytical treatment, the Monte Carlo method (Section 2.2.3) was employed to determine the relationship between the measured pressures and the effective capture coefficient of the titanium surface. In the numerical analysis a cylinder was used for the program which had the same dimensions as the test cell. The chamber was divided into a pumping half and a nonpumping half, and the midplane between the two halves was subdivided into an imaginary 18 by 24 grid as illustrated by Fig. 7. The effect of collisions with any obstructions in the chamber (titanium sources and shields, for example) was also taken into account.

The particle fluxes passing through each of the small grid areas toward and away from the pumping surface were computed for various values assigned for the capture coefficient using the general technique described in Section 2.2.3. Since the gas and pumping surface were at nearly the same temperature, multiple collisions would not alter the value of the capture coefficient by energy accommodation for these tests; therefore, the capture coefficient was considered to remain constant.

A typical result of the computer program is presented in Fig. 8 in the form of the total number of molecules passing through each of the small grid areas from the nonpumping surface to a pumping surface, which, in this case, had an assigned capture coefficient of 0.5. Similar information is given in Fig. 9 for the molecules which traverse the midplane passing from the pumping surface to the nonpumping surface (i. e., the flux return). The ratio of impinging gas flux to rebounding flux corresponds to the pressure ratio that would be obtained by the gage for a given capture coefficient. This pressure ratio can be obtained by taking the ratio of the number of molecules which pass through some area of the midplane from the nonpumping surface to those which pass through the same area from the pumping surface. The results shown in Figs. 8 and 9 represent several hours of computer time. Since the computer was not available for any longer period, several maps such as shown in Figs. 8 and 9 have been added together to obtain a better statistical average. This addition process was carried out for several sample areas (shown by the boxes superimposed in Figs. 8 and 9) rather than the entire map until the value of flux ratio or pressure ratio reached a constant value. Figure 10 illustrates how many molecules had to be considered in summing the calculations before the proper value of the pressure ratio was achieved for the case of an assumed surface capture coefficient of 0.40. This procedure was repeated for various values of capture coefficient. The resulting relationship between capture coefficient and pressure ratio for the chamber and pump geometries employed in these tests is given in Fig. 11. Capture coefficients obtained from these tests were determined from the measured pressure ratio by use of Fig. 11.

This numerical technique was also employed to determine the uniformity of the particle flux across the midplane for the purpose of positioning the pressure gage. In order to test for uniformity at the midplane, sample areas of several maps were added together (the five blocks enclosed by the solid lines in Figs. 8 and 9). The number of particles passing through these areas were 13,890; 13,775; 13,878; 14,151; and 14,029, resulting in a maximum variation of about 2 percent. Inspection of Figs. 8 and 9 does reveal an apparent decrease of particle flux around the edges, but this is believed to be caused by a

numerical analysis inaccuracy in the program. In any case the flux rates are uniform over the center portion of the midplane, and the pressure gage placement was not critical.

It was also of interest to determine the effect of the blockage caused by the filament shields. To do this, the program was run with and without the shields, and no significant difference was found in the flux ratio.

SECTION V RESULTS AND DISCUSSION

The experimental values obtained for the effective capture coefficient, at a substrate temperature and gas temperature near 20°C, are shown in Fig. 12. The effective capture coefficients were determined from the pressure ratio obtained from the rotating gage technique. The results of the Monte Carlo calculations for the cylindrical test chamber (Fig. 11) were utilized to relate the measured pressure ratio to the capture coefficient. Capture coefficients are plotted as a function of the ratio of titanium atoms deposited per unit time per unit area to the number of hydrogen molecules impinging on the substrate per unit time per unit area, that is, the value α .

These data have also been used to evaluate the unknown factors which appear to Eq. (37). The basic capture coefficient of a titanium surface (C) may be obtained by noting that as α becomes large

$$\lim_{\alpha \rightarrow \infty} \psi(\alpha, C) = \lim_{\alpha \rightarrow \infty} \frac{\alpha C}{KC + \alpha} = C \quad (38)$$

From the experimental data, it is seen that the effective capture coefficient did reach a limiting value of about 0.12. Using this value for C, the value of K was determined by a trial and error process to provide a good fit with the experimental data. The curve shown in Fig. 12 was obtained for K = 1. This curve indicates that each titanium atom adsorbed one hydrogen molecule, which is in agreement with Clausing's findings (Ref. 2). The trend of the data also agrees with Clausing's results; however, because of the differences in experimental procedure and measurement technique, a direct comparison of the values for capture coefficient is probably not valid.

The present data agree well with the proposed simple theoretical model, which assumed that the sorption process, during continuous deposition, is primarily a surface interaction of the titanium atoms with the impinging hydrogen molecules. This process is probably dominant

at high values of α . However, other sorption processes are possibly taking place; at very low values of α , diffusion or migration of hydrogen into the titanium may be dominant. A discussion of a diffusion mechanism is presented by Kindall (Ref. 3).

Kindall has also found that the capture coefficient is a strong function of gas and substrate temperature. It might be expected, then, that the value of 0.12 for capture coefficient would be significantly different at a different temperature.

It can be seen from Fig. 12 that the effective capture coefficient increased rapidly with increasing titanium deposition rate until the titanium deposition rate equaled the gas impinging rate. Beyond this, little was gained by increasing the titanium deposition, so that, in practice, for a substrate at room temperature and a given hydrogen gas load, the titanium deposition rate should be no less than the hydrogen gas impinging rate.

SECTION VI CONCLUSIONS

The following conclusions may be drawn from the results of this research:

1. The model assumed for deriving the functional dependence of the effective capture coefficient with titanium evaporation rate and hydrogen gas load leads to an equation which has the same form as the experimental data.
2. The results demonstrated that there was little to be gained by evaporating more than one atom of titanium for each hydrogen molecule.
3. One titanium atom adsorbed no more than one hydrogen molecule.
4. The rotating gage technique is a simple and reliable method for measuring capture coefficients, particularly if a hemispherical pumping surface is used.

APPENDIX I INVARIANCE OF CAPTURE COEFFICIENT MEASUREMENT WITH GAS TEMPERATURE

When a gage is calibrated, it is calibrated for a given gas temperature. If a different temperature is experienced in operation, it is necessary that this calibration be corrected by the relation for thermal transpiration (Ref. 6).

$$P_c = P_g \sqrt{T_c/T_g} \quad (\text{I-1})$$

where

P_c = cell pressure

P_g = gage pressure

T_c = cell temperature

T_g = gage temperature

This requires knowing the gas temperature, which is often difficult to determine, but, again, with the rotating gage this problem can be eliminated. The flux entering the gage must be equal to the flux leaving the gage. For the impinging molecules then

$$\left(\frac{dn}{dt} \right)_i = \frac{P_i N_o a}{\sqrt{2 \pi M R T_i}} = \frac{P_{gi} N_o a}{\sqrt{2 \pi M R T_g}} \quad (\text{I-2})$$

where

P_{gi} = pressure in the gage caused by the
impinging flux

R = gas constant

M = molecular weight of the gas

T_g = temperature of the gas in the gage

T_i = temperature of the impinging gas

and the rebounding flux will be

$$\left(\frac{dn}{dt} \right)_r = \frac{P_r N_o a}{\sqrt{2 \pi M R T_r}} = \frac{P_{gr} N_o a}{\sqrt{2 \pi M R T_g}} \quad (\text{I-3})$$

where

P_{gr} = pressure in the gage caused by the
rebounding flux

T_r = temperature of the rebounding flux

As before,

$$\left(\frac{dn}{dt}\right)_r = (1 - C) \left(\frac{dn}{dt}\right)_i \quad (\text{I-4})$$

so that

$$\frac{P_{g_r} N_o a}{2 \pi MRT_g} = \frac{(1 - C) P_{g_i} N_o a}{2 \pi MRT_g} \quad (\text{I-5})$$

$$P_{g_r} = (1 - C) P_{g_i} \quad (\text{I-6})$$

and is, therefore, independent of cell temperature.

APPENDIX II

ELIMINATION OF THE CALIBRATION OF IONIZATION GAGES BY USE OF THE ROTATING GAGE TECHNIQUE

It is not necessary that the ionization gage be calibrated, nor will a change in gage sensitivity affect the results. This can best be understood by reviewing the operation of a typical ionization gage (see sketch below). Electrons are emitted from the hot filament; in their travel to the grid, they will produce a certain number of ions. The ion current, I , produced will be proportional to the electron emission current, I_e , and the gas density or pressure in this region, that is,

$$I = k P I_e \quad (\text{II-1})$$

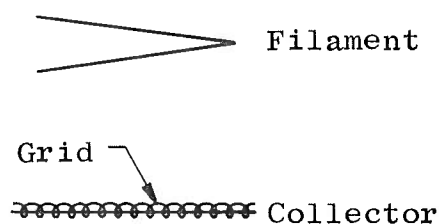
The gage is then calibrated by adjusting I_e so that

$$k I_e = 1 \quad (\text{II-2})$$

As can be seen from Eq. (6), with the rotating gage method, this calibration is unnecessary since for Eq. (6),

$$C = \left(1 - \frac{P_r}{P_i} \right) \quad (\text{II-3})$$

$$= \left(1 - \frac{I_r k I_e}{I_i k I_e} \right) = 1 - \frac{I_r}{I_i} \quad (\text{II-4})$$



APPENDIX III AREA VIEWED BY A TUBULATED GAGE

To determine the area viewed by an ionization gage, first consider a uniform gas flux impinging on a thin-walled orifice. The number of molecules, dn , which enters the orifice through a cone of solid angle ω and of half-vertex angle θ will be

$$dn = n_z \cos \theta \, d\omega \quad (\text{III-1})$$

where

n_z = number of molecules impinging from the direction
of the normal

$$d\omega = 2\pi \sin \theta \, d\theta \quad (\text{III-2})$$

$$n_t = \int_0^{\pi/2} 2\pi n_z \sin \theta \cos \theta \, d\theta \quad (\text{III-3})$$

Where n_t is the total number that enters,

$$n_z = \frac{n_t}{\pi} \quad (\text{III-4})$$

Integrating Eq. (I-3), the number entering the cone defined by θ will be

$$n_z = n_t \sin^2 \theta \quad (\text{III-5})$$

so that the percent of the total number of molecules γ which will enter the orifice through the cone will be

$$\gamma = \frac{n_z}{n_t} 100 = 100 \sin^2 \theta \quad (\text{III-6})$$

This is shown graphically in Fig. III-1. It can be seen that 90 percent of the molecules which enter the orifice come from within an angle of 72 deg.

Now consider an ionization gage contained in an envelope such as shown in Fig. 1. The number of molecules which enters the gage as a function of the angle was determined by use of a computer program employing the Monte Carlo Method. The results of this program for the gage used in this experiment are also shown in Fig. III-1. It can be seen that the conductance of the tube has served to narrow the viewing angle of the gage, and now 90 percent of the molecules which enter the gage come from within a cone of 65 deg.

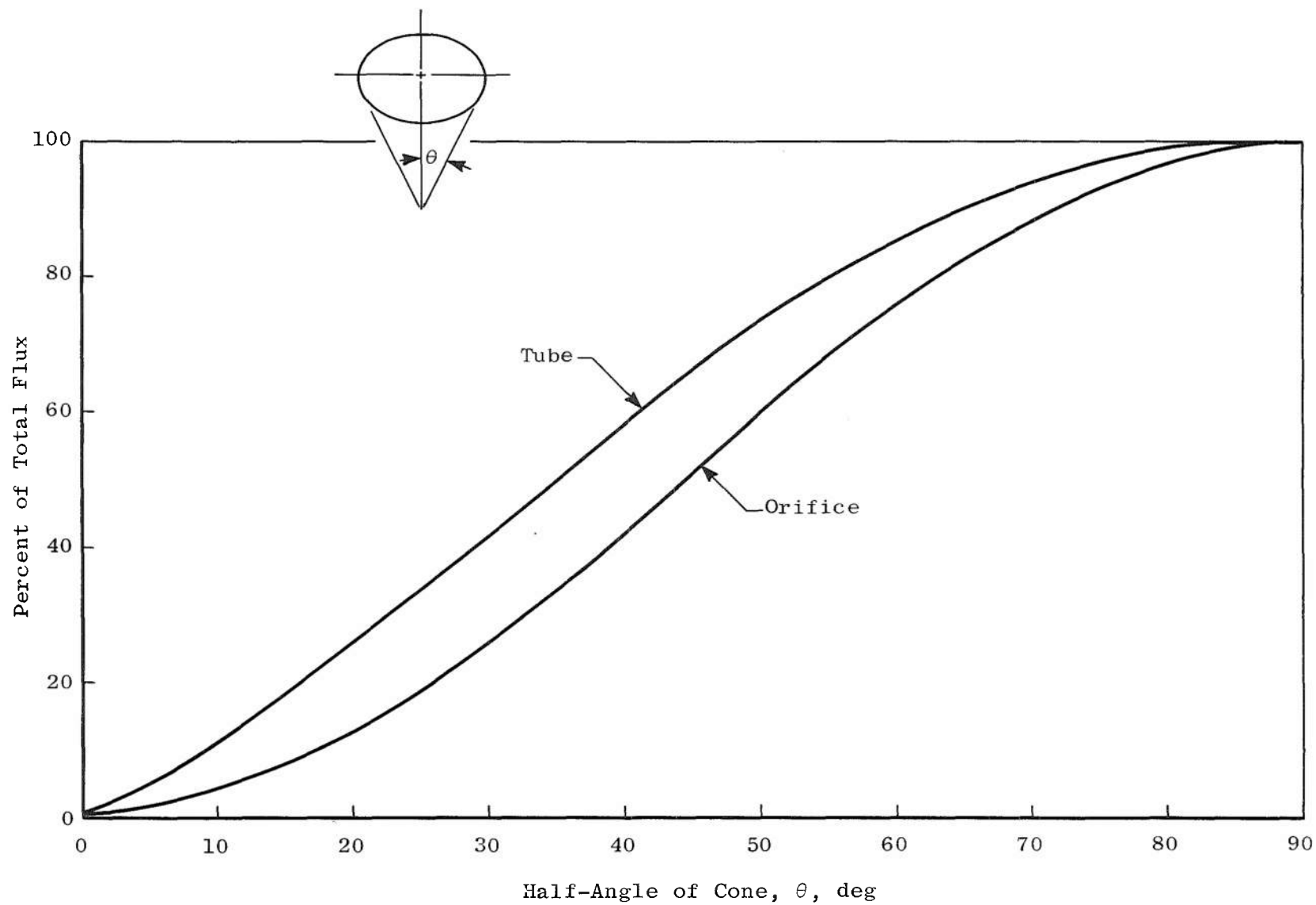


Fig. III-1 Percent of Total Number of Molecules which Enter a Cone of θ deg

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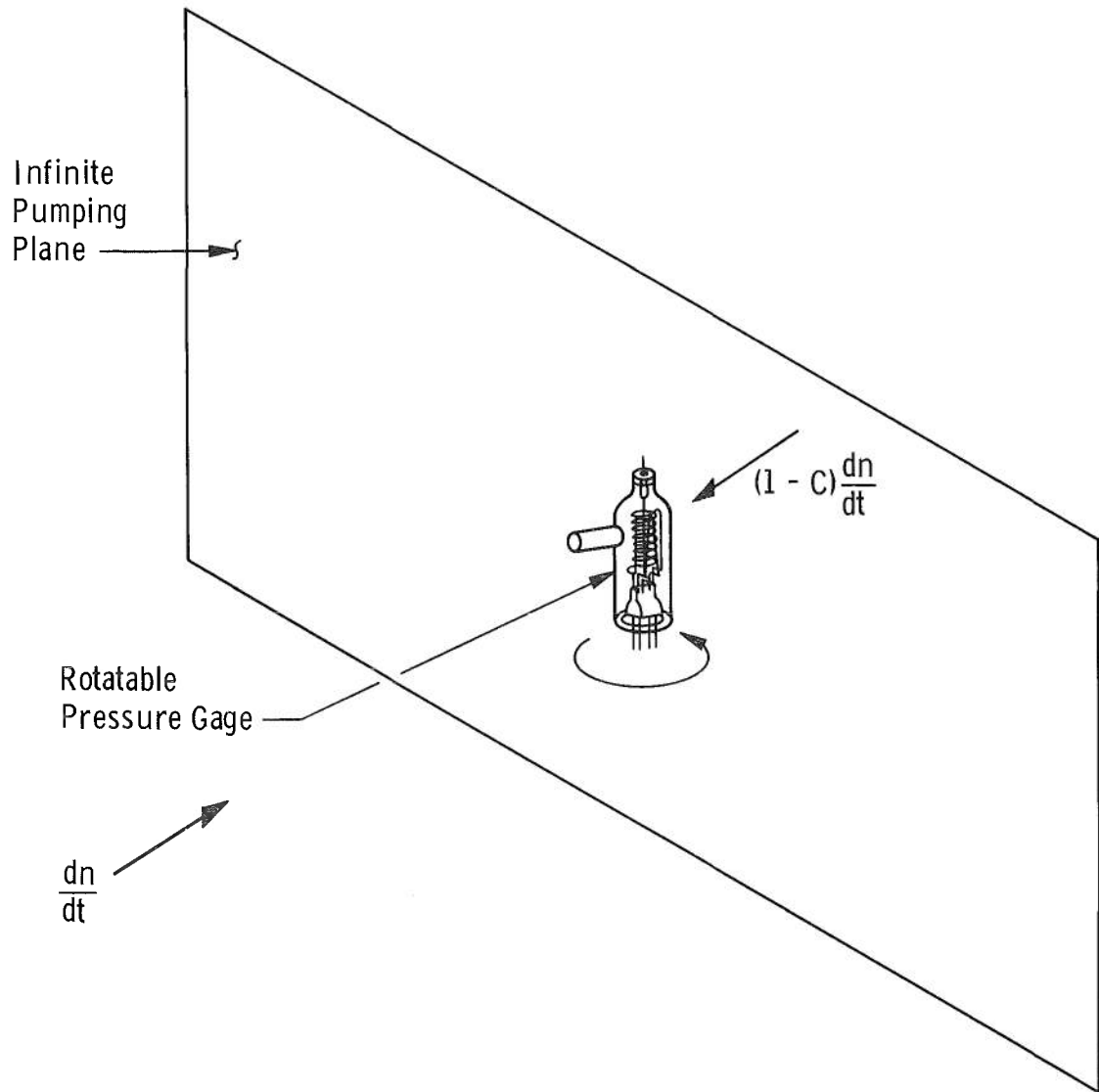


Fig. 1 Infinite Pumping Plane

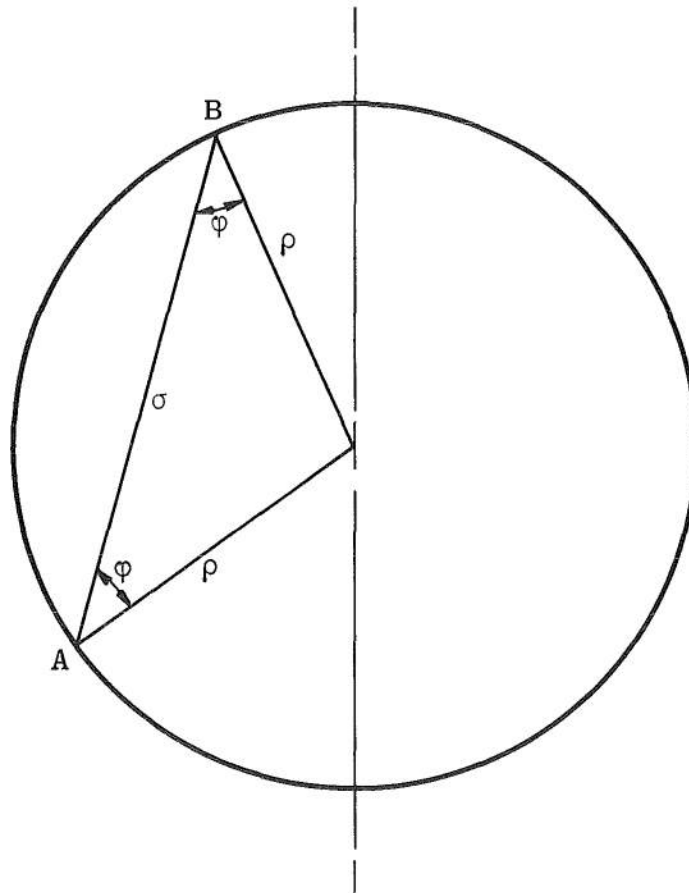


Fig. 2 Sketch Used for Derivation of Eq. (9)

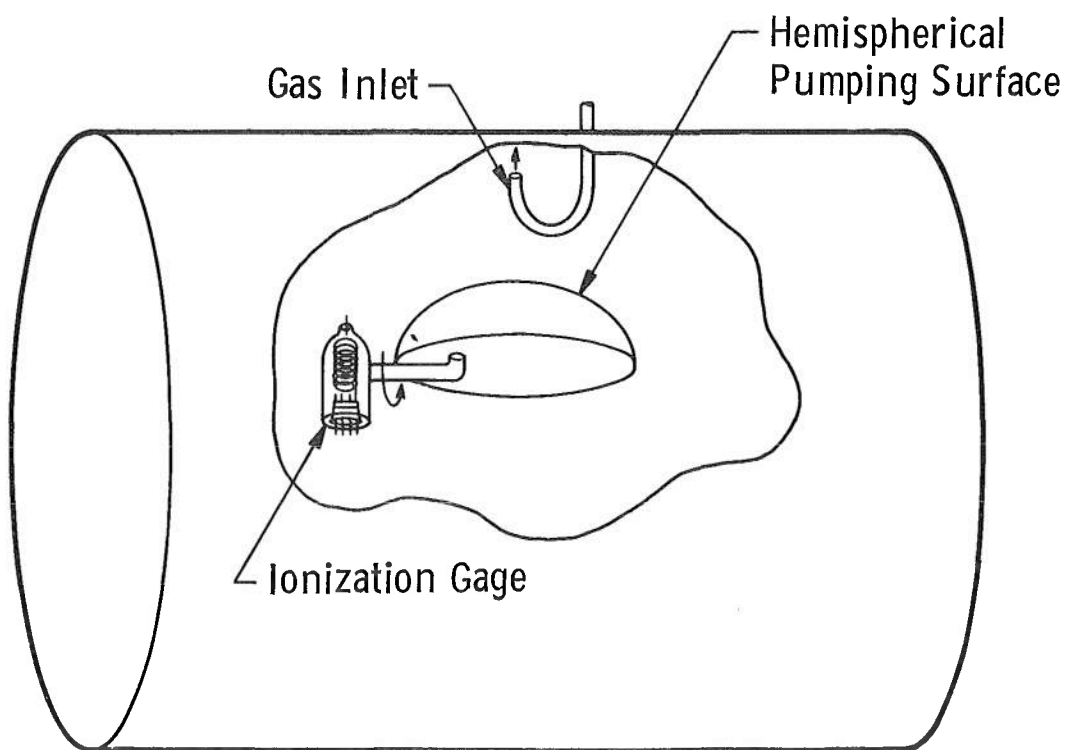


Fig. 3 Schematic of Vacuum Chamber with a Hemispherical Pumping Surface

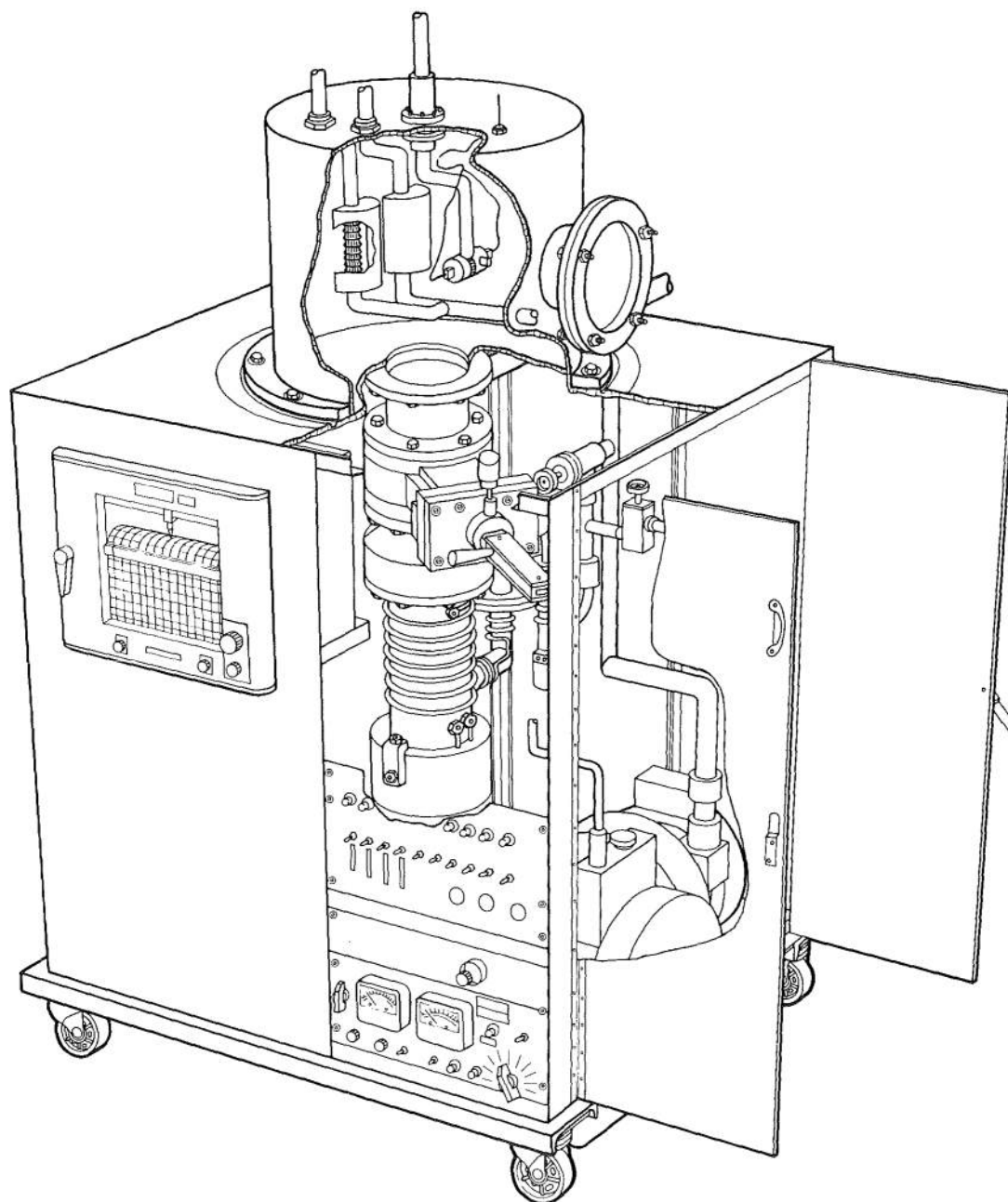


Fig. 4 Vacuum System

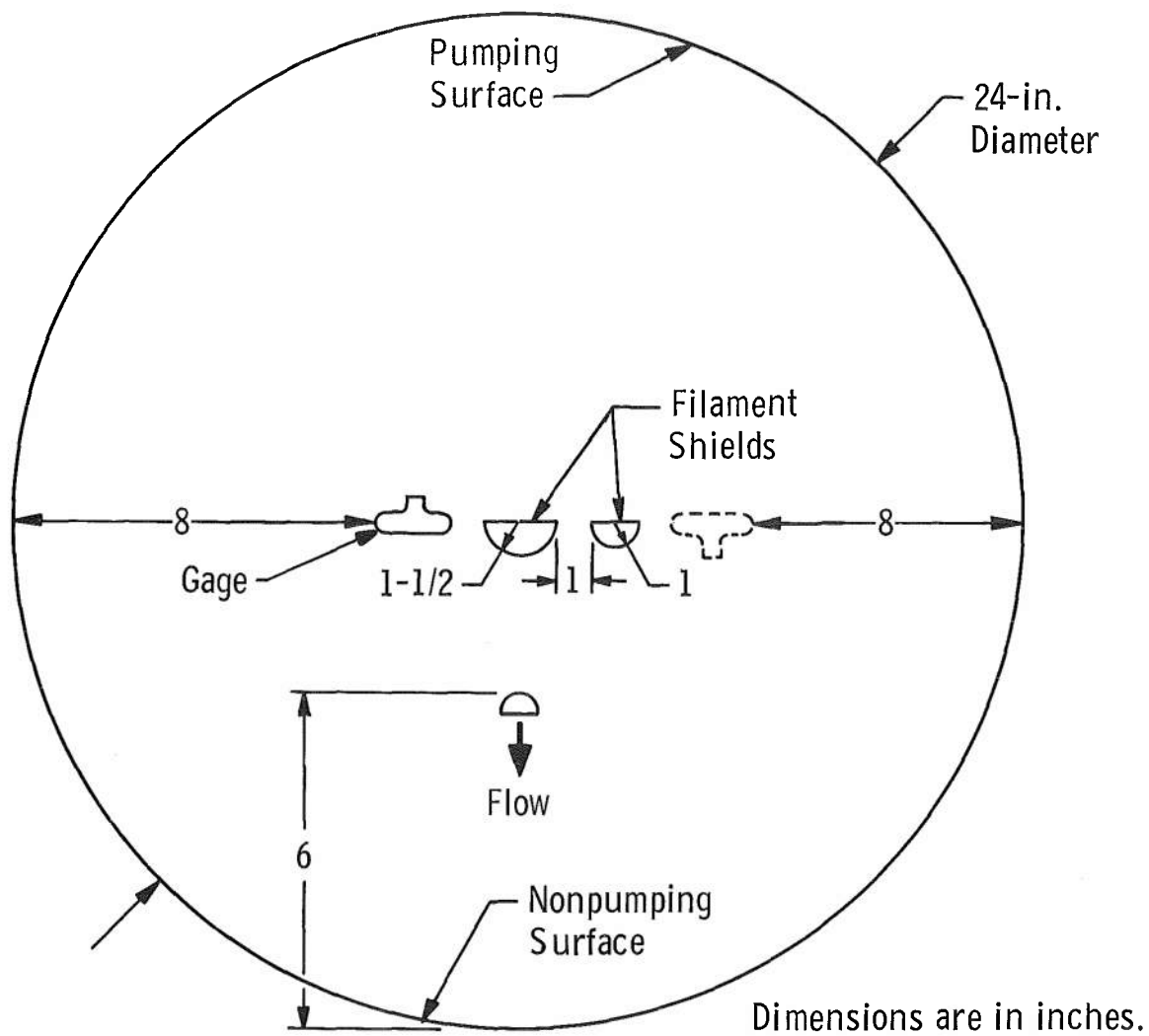


Fig. 5 Schematic of Cylindrical Vacuum Chamber Showing Location of Filament Shield and Rotating Gage

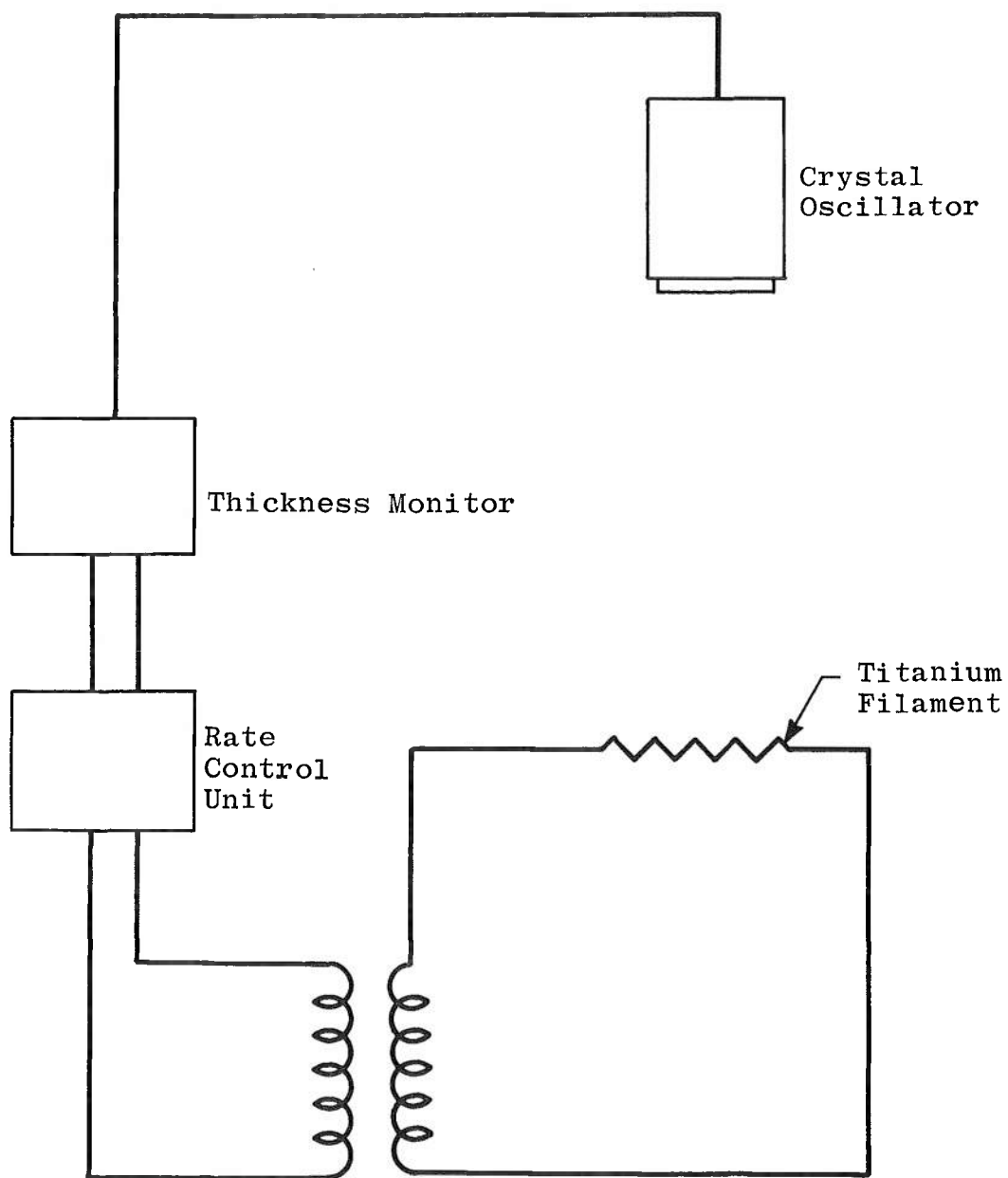


Fig. 6 Schematic of Evaporation Monitor and Control Unit

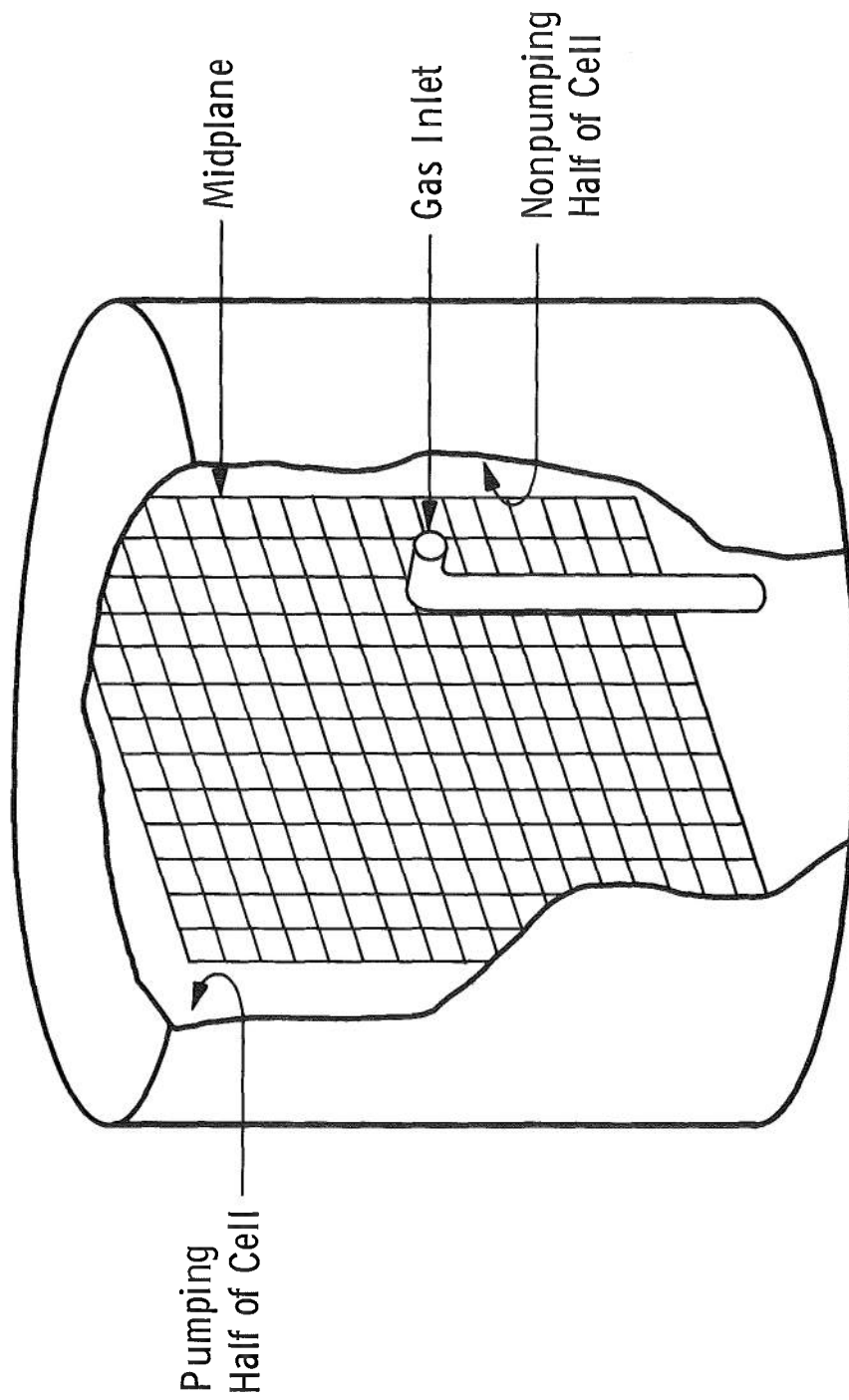


Fig. 7 Position of Midplane in Vacuum Cell

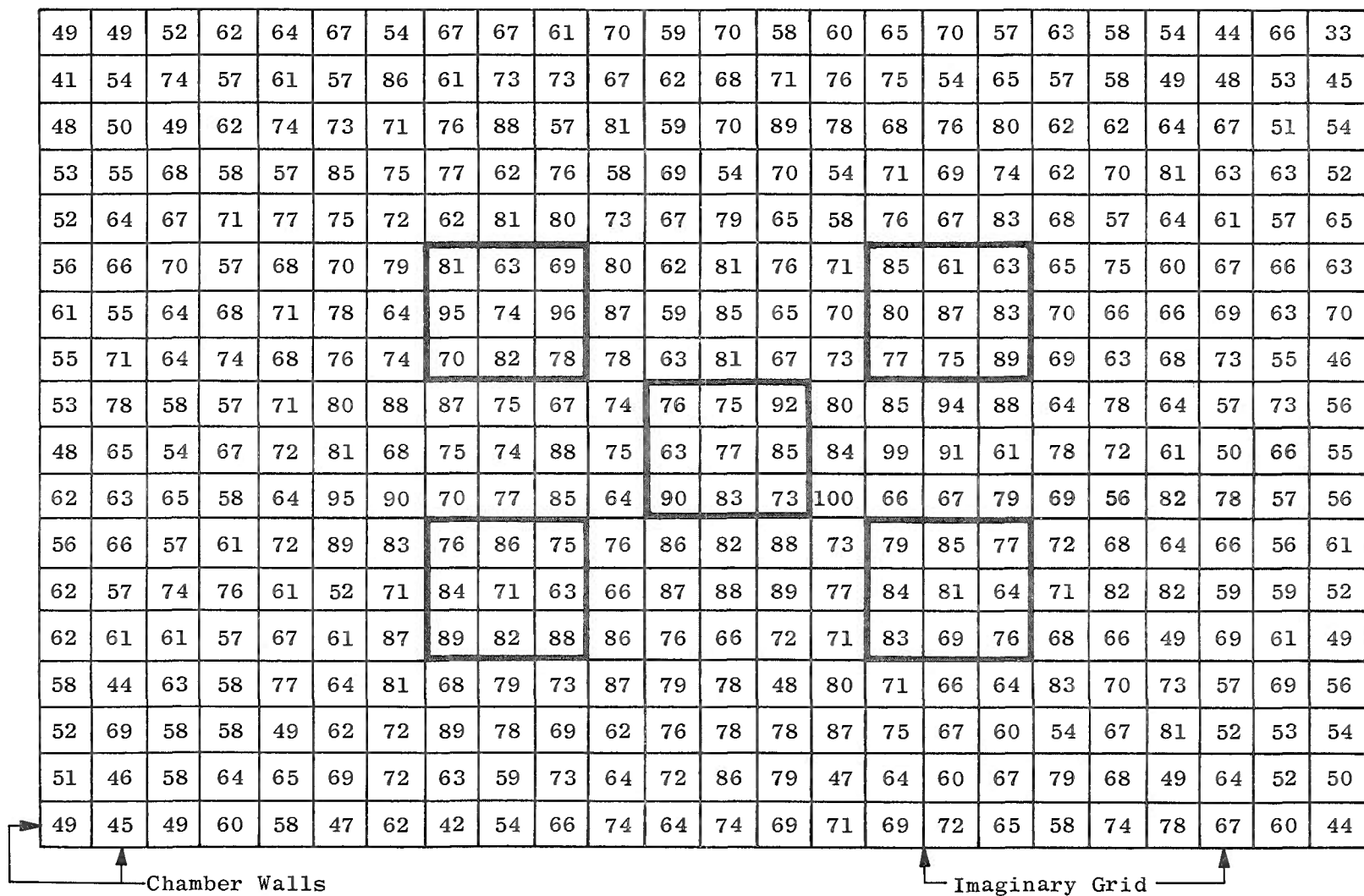


Fig. 8 Midplane Flux to Pumping Surface

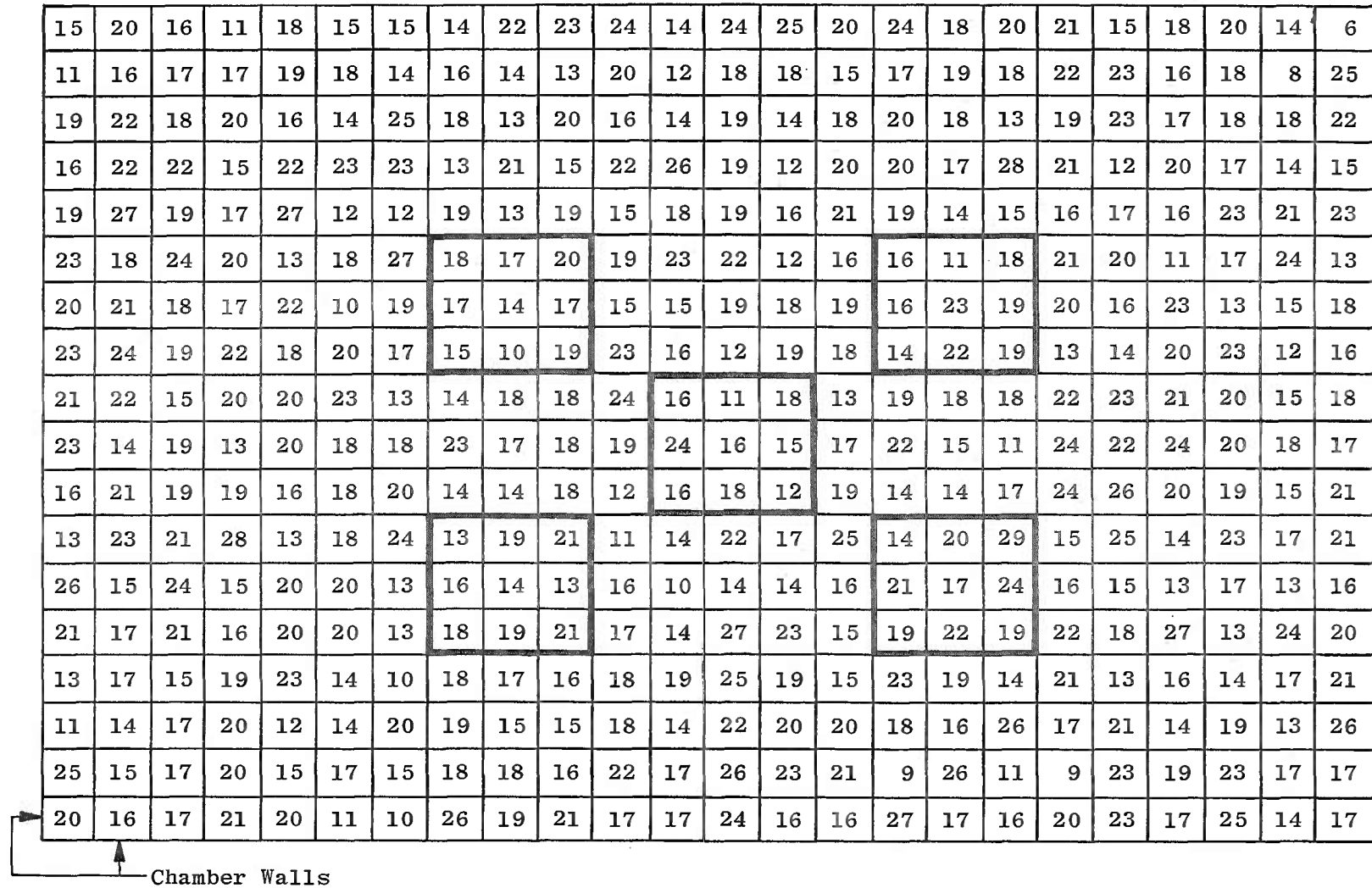


Fig. 9 Midplane Flux from Pumping Surface Assuming Capture Coefficient of 0.5

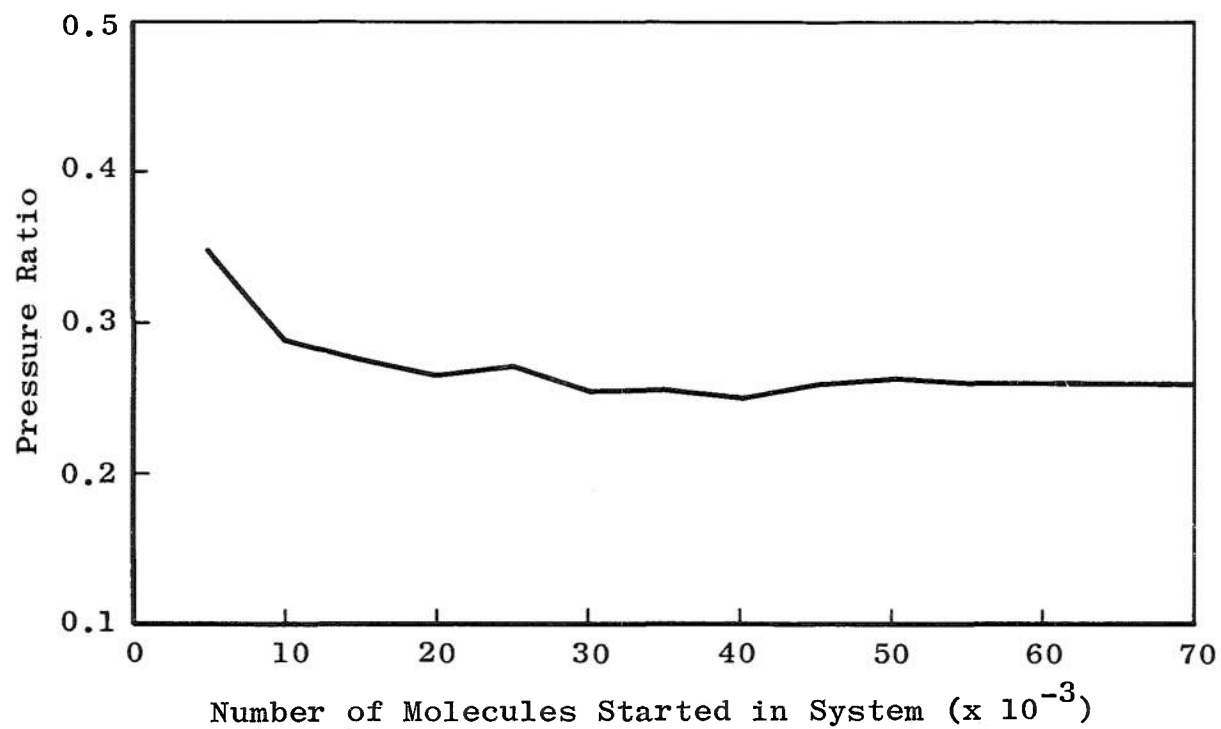


Fig. 10 Stability of Computed Capture Coefficient for a Cylindrical Chamber

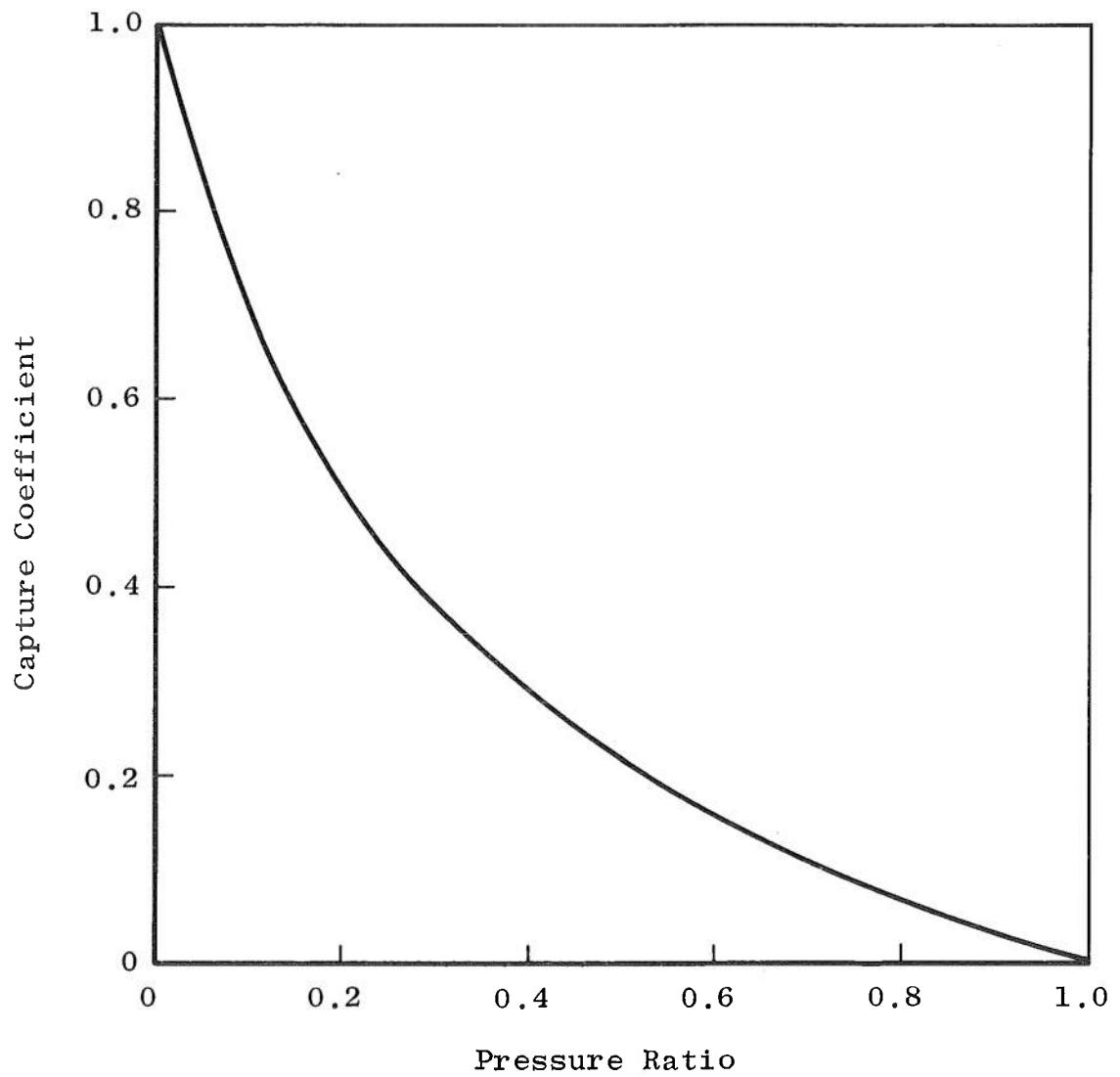


Fig. 11 Capture Coefficient as a Function of Pressure Ratio for a Cylindrical Chamber

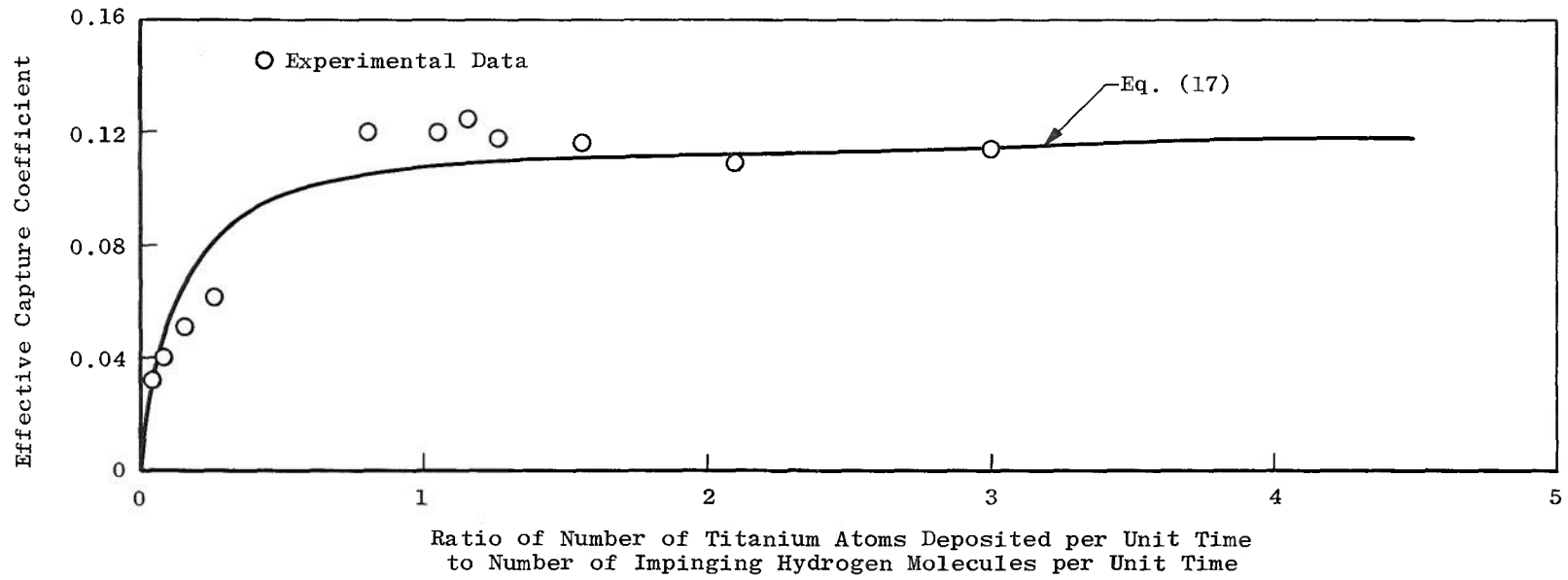


Fig. 12 Comparison of Theoretical and Experimental Capture Coefficients

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